



Case 4233

#4
H.J.J
12-12-9

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

the Application of

DONALD B. APPLEBY
DAVID J. BRUNO, JR.
PATRICK J. CORRIGAN
JOHN K. HOWIE
JU-NAN KAO
SCOTT D. PEARSON
RICHARD G. SCHAFERMEYER
GLEN R. WYNESS

RECEIVED
DEC 8 1990
GROUP 180

Serial No. 07/580,706

: Group Art Unit 183

Filed September 11, 1990

: 18X White
12-3-90

POLYOL POLYESTER SYNTHESIS

:

SUBMISSION OF INFORMATION

The Commissioner of Patents and Trademarks
Washington, DC 20231

Dear Sir:

The following information is being submitted herewith for consideration by the Examiner in the examination of the above-named application. This submission does not constitute either a representation that a thorough search has been made or an admission that the references cited herein are properly citable against the above-captioned patent application. Comments accompanying each reference are believed to constitute a fair representation of the pertinency of said reference. However, those comments are not intended to take the place of the Examiner's independent consideration of each reference.

Of the references known to the applicants, the most pertinent are believed to be:

U.S. Patent 2,893,990, Hass et al., issued July 7, 1959, describes producing lower (less than about 3 fatty acyl groups) polyol fatty acid esters,

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to:
Commissioner of Patents & Trademarks,
Washington, D.C. 20231,

on 3 December 1990

Robert B. Aylor

Registration No. 20,369

U.S. Patent 2,948,717, Babayan et al., issued Aug. 9, 1960, discloses a method for purifying sugar esters.

U.S. Patent 3,251,827, Schnell et al., issued May 17, 1966, discloses the preparation of lower sugar esters.

U.S. Patent 3,558,597, von Brachel and Schon, issued Jan. 26, 1971. This patent discloses using potassium stearate and potassium carbonate in a process to make sucrose monoester.

U.S. Patent 3,600,186, Mattson and Volpenhein, issued August 17, 1971. This patent discloses fatty acid (8-22) polyesters of sugar or sugar alcohols (SPE) as low calorie fats. The disclosed process for making erythritol tetraester of olive oil fatty acids is a laboratory process involving the use of solvents.

U.S. Patent 3,644,333, Osipow et al., issued Feb. 22, 1972, discloses transesterification in the presence of a "micro emulsion."

U.S. Patent 3,792,041, Yamagishi et al., issued Feb. 12, 1974, discloses preparation of sucrose polyesters.

U.S. Patent 3,963,699, Rizzi and Taylor, issued June 15, 1976. This patent discloses batch, solvent-free processes for making SPE comprising an initial stage of reacting sugar or sugar alcohol with a fatty acid alkyl ester in the presence of a soap and a strongly alkaline catalyst like an alkali metal alkoxide. In a later stage, additional ester is added to obtain more completeness. There is no discussion about the purity or form of the various reactants and catalysts. There is no indication of how much sucrose octaester is formed. Bleaching clay and solvent treatments are used for clean-up. The products contain relatively large amounts of methyl esters, but are stated to be usable as is.

U.S. Patent 4,005,196, Jandacek et al., issued Jan. 25, 1977, discloses fortifying SPE with vitamins.

U.S. Patent 4,032,702, James, issued June 28, 1977. This patent discloses the preparation of sucrose surface active agents from sucrose and alkyl fatty acid esters by not removing the alcohol that is formed. Methyl stearate, K_2CO_3 , and potassium stearate are used in Example I.

U.S. Patent 4,334,061, Bossier, issued June 8, 1982, discloses purifying SPE's.

U.S. Patent 4,517,360, Volpenhein, issued May 14, 1985. This patent discloses the use of potassium, sodium or barium carbonate as catalysts for making SPE. There is no disclosure of purity or physical form of the reactants in the interesterification step and no disclosure of any specific clean-up procedure. The generic disclosure suggests there is no problem with obtaining cleaned-up sucrose esters. Degrees of completion to octaester of up to 85% are disclosed, but not the level of color/odor materials, alkyl/esters, etc., in the finished product.

U.S. Patent 4,518,772, Volpenhein, issued May 21, 1985, contains essentially the same disclosure as the '360 patent. The claims are limited by the molar ratio of soap to polyol used in the initial stage (0.6:1 to 1:1).

The following European Patent Applications disclose several modifications of polyester syntheses:

0,235,836, Bodor et al., published Sept. 9, 1987, discloses using trans-unsaturated fatty acids for improved oxidative stability.

0,256,585, Van der Plank et al., published Feb. 24, 1988, discloses premixing polyol with alkaline "catalyst".

0,301,634, Van der Plank, published Feb. 1, 1989, discloses using shorter chain fatty acid soaps as emulsifiers.

0,315,265, Van Nispen et al., published May 10, 1989, discloses using "high shear" to form a homogeneous dispersion of the reactants.

0,319,091, Van Lookeren, published June 7, 1989, discloses reducing alkali metal content to less than 5 ppm by weight.

0,319,092, Van Lookeren, published June 7, 1989, discloses water washing the SPE at a pH of at least 12.5

0,320,043, Willemse, published June 14, 1989, discloses spray drying at least part of the reactants for homogeneity.

0,322,971, Willemse, published July 5, 1989, discloses a two stage process with lower partial esters in the first stage as an emulsifier.

0,323,670, Meszaros Grechke et al., published July 12, 1989, discloses removing soap emulsifier when the reaction is 15% to 60% complete.

0,349,059, Willemse, published Jan. 3, 1990, discloses stripping alcohol at the end of the reaction.

Jap. Pat. Appln. 61-106589, Kinami, published May 24, 1986. This application discloses a process for making sucrose polyesters using a lower sucrose polyester as the compatibilizing agent for the sucrose, the short chain alkyl fatty acid ester and the catalyst. Only unpurified products are disclosed. This application teaches that granular sugar can be used.

Jap. Pat. Appln. 51-14486, Yamagishi et al., published May 10, 1976. This application discloses using mixtures of inorganic "hydrophobic," e.g., K_2CO_3 , and organic, e.g., $KOCH_3$, catalysts for improved sucrose mono-fatty-acid ester production.

Brit. Pat. 1,399,053, Parker et al., published June 25, 1975. This patent discloses reacting sucrose and triglycerides at lower temperatures (110-140°C) to form a surface active agent.

Literature references which provided background and many process details include:

Sugar Esters, Osipow et al., The Journal of the American Oil Chemists' Society (JAOCs), Vol. 34, pp. 185-188, April 1957.

Preparation of Sucrose Esters by Interesterification, Feuge et al., JAOCs, Vol. 47, pp. 56-60, February 1970.

Nebraska-Snell Process, Speech by Kammerlohr.

A Solvent-free Synthesis of Sucrose Polyester, Rizzi and Taylor, JAOCs, Vol. 55, pp. 398-401, April 1978.

Sucrose Ester Surfactants - A Solventless Process and the Products Thereof, Parker et al., Sucrochemistry (Publication details not available at this time.)

Art relating to making partial esters and/or processes that utilize a solvent has, in general, been excluded.

Copies of the above documents are submitted herewith. The translations are believed to be accurate but are not warranted as such.

The Examiner is requested to acknowledge receipt and consideration of these documents in the next action on this application. A completed copy of PTO 1449 is attached hereto.

Respectfully submitted,

DONALD B. APPLEBY ET AL.

Cincinnati, Ohio
December 3, 1990
(513) 626-3713
RBA/bh/bpto/177

By Robert B. Aylor
Robert B. Aylor
Attorney for Applicants
Registration No. 20,369